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13. ABSTRACT (Maximum 200 words)

The purpose of chemical protective clothing is to shield or isolate individuals from the chemical, physical, and biological hazards that may be encountered in potentially hazardous environments. There is increased interest in the development of protective wear that provides protection against chemical and biological threats while also being lightweight, comfortable, stretchable and affordable. For this purpose, the melt blown (MB) processing of thermoplastic polyurethanes (TPUs) was studied using the 6-inch and 20-inch MB lines at TANDEC, University of Tennessee (UT). This study was performed in three phases. The objective of the Phase 1, which was conducted first on the 6-inch MB line and then on the 20-inch MB line, was to establish an envelope of MB operating conditions versus web performance properties with three Noveon TPUs having different breathability and elasticity properties. The objective of Phase 2 was to use this information to optimize processing conditions on the 20-inch MB line through the evaluation of the web barrier and strength properties. Phase 3 involved the PI (Wadsworth) supervising a consultant, Bernard L. Beals, Hamilton, MT, in designing a MB line more suitable for processing TPUs fed by a twin-screw extruder to allow the incorporation of additives along the length of the extruder, and especially just before the MB die. Beals was also utilized to inspect the twin-screw extruder which Wadsworth located at Shell Chemical Company, Houston, TX, to determine if it was suitable for the MB process before UT accepted the donation.

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Meltblowns for Chemical Protective Liners

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ABSTRACT

The purpose of chemical protective clothing is to shield or isolate individuals from the chemical, physical, and biological hazards that may be encountered in potentially hazardous environments, including biological hazards. When dealing with hazardous materials or working in toxic environment, chemical protective clothing is critical to guard against the effects of toxic products, which could enter the body through inhalation or skin absorption, or cause tissue damage upon contact with the skin. So, there is increased interest in the development of protective wear that provides protection against chemical and biological threats while also being lightweight, comfortable, stretchable and affordable. For this purpose, the melt blown (MB) processing of thermoplastic polyurethanes (Noveon Estane® 58237, 58245 and 58280) was studied using the 6-inch and 20-inch MB lines at TANDEC, University of Tennessee, Knoxville. This study was performed in three phases. The objective of the Phase 1, which was conducted first on the 6-inch MB line and then on the 20-inch MB line, was to establish an envelope of MB operating conditions versus web performance properties with three Noveon TPUs having different breathability and elasticity properties. The objective of Phase 2 was to use this information to optimize processing conditions on the 20-inch MB line through the evaluation of the web barrier and strength properties. Another focus of the Phase 2 was to determine the extent to which polymer throughput rate could be increased, while maintaining acceptably small fiber diameters and other key performance attributes to improve processing economics. The MB fabrics were tested for basis weight, thickness, air permeability, and tearing/tensile strength. Average fiber diameters were determined by optical microscopy. Furthermore the effect of thermal point calendering on tensile and tearing strength was determined on two MB fabrics made from Estane® 58245.

Phase 3 of this STIR project involved the PI (Wadsworth) supervising a consultant, Bernard L. Beals, Beals Consulting, Hamilton, MT, in designing a High Performance Melt Blown (HPMB) Line with die width of 10 inches, which was to be fed by a twin-

screw extruder to allow the incorporation of additives along the length of the extruder, and especially just before the MB die. An objective of this phase was to design a MB die more suitable for processing TPUs and other high viscosity/high performance polymers. Beals was also utilized to inspect the twin-screw extruder which Wadsworth located at Shell Chemical Company, Houston, TX, to determine if it was suitable for the MB process before the University of Tennessee accepted the donation.

BACKGROUND

Although the consumption of thermoplastic polyurethane (TPU) elastomers is still much less than conventional polyurethanes which must be solvent spun to produce fibers, the fact that TPUs may be melt spun into fibers makes them much more versatile. However, melt spinning elastic fibers is difficult because of their tendency to snap back during attenuation of the spinline. This is even more challenging with melt blowing since the filaments are attenuated by air skin friction and by air form drag, and the filaments may be discontinuous and are not positively held by a take-up spindle or nip while in flight to the collector. Yet MB nonwovens have an inherent advantage over spunbond (SB) nonwovens and other fabric made from conventionally spun polyurethane fibers in that MB fabrics typically have average fiber diameters ranging from 2-6 μm compared to 12-50 μm with SB webs and conventional textiles. Thus, if MB TPUs can be produced with average fiber diameters in the range of 2-6 μm, they will provide much better filtration and barrier protection, and with hopefully acceptable breathability. Furthermore, TPUs have many inherent advantages such as good hardness for any given modulus, high abrasion and chemical resistance, excellent mechanical and elastic properties, and also have hydrophilic compatibility.

Towards this end, preliminary melt blowing (MB) studies of thermoplastic polyurethane (Noveon Estane® 58277) were performed on February 7, 2001 on the 6-inch line at TANDEC, The University of Tennessee, Knoxville, before this Short Term Innovative Research (STIR) Project No. DAAD19-01-1-0732 was approved and conducted during 6 August 2001 - 5 February 2002. The 6-inch MB line was used in the preliminary study with a minimal 10 pound quantity of the thermoplastic polyurethane (TPU), although edge effects reduced the useable width of the web produced for testing. It was anticipated that melt processing of TPU would be difficult since melt blowing can be particularly challenging due to the elasticity of the filaments. Therefore the first study funded by TANDEC was undertaken to identify processing parameters and scale up guidelines for future processing. MB TPU webs were produced with light, medium and heavy average basis weights of 0.7, 2.2 and 4.9 oz/yd^2 (23.9, 75.4 and 164.9 g/m^2), three different air flow rates and at die-to-collector distances of 10, 12, 14, 18 and 24 in. The MB spinneret with orifice diameters of 0.020 inch, 10/1 L/D, and with 20 holes per inch was used. The air knife slot widths on each side of the 60 degree angle nose tip was 0.060 in. and setback of the nose tip from the inside edge of the air knives was also 0.060 in. The polymer throughput rate was 0.48g/hole/min. Die temperature was held at 400°F while air temperature in the blower was varied from 398°F to 498°F. Web collection speeds varied from 2 to 16 ft/min. Melt pressures after the metering pump varied from 178 to 308 psig. Die air pressures ranged from 7 to 14 psig, depending on the desired volume

of air flow to the die. All fabrics were tested for basis weight, thickness, air permeability, and tearing and tensile strength. Selected fabrics were also evaluated for the effect of thickness upon moisture vapor transmission rates, to optimize breathability for this application.

The research findings from the preliminary study were presented by Larry Wadsworth at the joint INDA/TAPPI International Nonwovens Technical Conference (INTC 2001) in Baltimore, MD during September 5-7, 2001.[1)] The authors of this paper were Larry C. Wadsworth, Christine (Qin) Sun, Dong Zhang and Rongguo Zhao of TANDEC and Heidi L. Schreuder-Gibson and Phil Gibson, U. S. Army Natick Soldier Systems Center. It was found that medium weight (~2 oz/yd^2) MB nonwoven fabrics of Estane thermoplastic elastomer exhibited the best balance of weight and strength, as well as acceptable air and moisture vapor transport properties. It also appeared from this earlier study that heavy weight (~5 oz/yd^2) nonwovens could be processed to maximize strength while minimizing resistance to air flow and moisture vapor diffusion.

FIRST PHASE OF STIR PROJECT

Process Conditions for Melt Blown Fabrics in First Phase

Per the recommendations of Dr. Heidi Schreuder Gibson, STIR Project Manager, and Susan Hemphill, Business Development Manager-Textiles, Thermoplastic Polyurethane, Noveon, Estane 58237, 58245, 58238 and 58280 pellets were ordered from Noveon. According to Susan Hemphill, Estane 58237 and Estane 58245 are from a similar class of Estane polymers, which have lower elasticity than the class to which Estane 58238 and 58280 belong, but the Estane 58237 and 58245 have higher breathability, and 58245 has higher breathability than 58237. She further noted that 58237 and 58245 were developed as breathable films to replace Gore-Tex® membranes; whereas, 58238 and 58280 were designed to replace Lyrca fiber[2)]. The four Estane TPUs were dried for 12 hr at 100 °C in a Conair Franklin Compu-Dry CD 60 hopper dryer to a moisture content <0.02%.

Results Obtained in First Phase of STIR Project

As shown in Table 1, the first six MB trials (Estane 58237, 58245, 58238 and 58280) in the STIR Project were made on the 6-inch MB line at TANDEC. For these trials, a MB spinneret with 0.020 in. diameter orifices, 10/1 L/D, and with 20 holes/in. was used. The air knife slot widths on each side of the 60 degree angle nose tip was 0.060 in. and setback of the nose tip from the inside edge of the air knives was also 0.060 inches. The polymer throughput rate was 0.5-0.7 g/hole/min. The die temperature was kept at 380 °F and the primary air temperature in the die was varied from 388-413 °F. There was concern by Noveon that die and air temperatures above 400 °F would result in polymer degradation and fiber strength loss[2)]. However, a continuous web could only be produced in Trial No. 1.3 on the 6-in. line with Estane 58237. The fabric weight was 150 /m^2, thickness was 0.616 mm and the air permeability was 202 ft^3/ft^2/min. However, the average fiber diameter as determined by optical microscopy was encouragingly low at 6.91 μm. The effective fiber diameter as determined by the method developed by Tsai[3)] using air permeability data was 12.86 μm.

The information gained from the trial runs on the 6-inch MB line described above was helpful in the next more successful series of MB trials in Phase 1 of the STIR on the 20-inch Accurate Products MB line at TANDEC. Compared to the 6-inch MB line which is primarily useful in screening polymers, the 20-inch MB line provide much more accurate and precise control of die and air temperatures and polymer throughput (20-inch MB line has Zenith-Nichols positive displacement gear pump and 6-inch line has no metering pump). The large-hole diameter die tip (0.018 in. orifice diameter, 4.5/1 L/D, and 20 holes/in.) was used. The air knife gap was 0.090 in. and the die tip setback was also 0.090 in. The polymer throughput rate was not determined, but is believed to have been less than 0.5 g/hole/min. As shown in Table 1, continuous webs were produced with Estane 58280 in Trials 2.1 and 2.3 resulting in basis weights of 71.25 and 100 g/m^2 and with Estane 58245, resulting in a basis weight of 17.5 g/m^2. In efforts to obtain finer fibers and softer, more uniform webs, the die temperature was increased to 390 °F and the primary air temperature was increased to 416-418 °F. However, both webs produced with Estane 58280 were rather coarse as was verified by the large fiber diameters, which were on the order of 19-20 μm, as determined both by optical microscopy and air flow measurements (effective fiber diameter). Nevertheless, it was encouraging that for the trials on the 20-inch MB line in Phase 1, an average optically determined fiber diameter of 5.42 μm was obtained with Estane 58245 and the web was quite uniform in appearance and had a much softer hand.

SECOND PHASE OF STIR PROJECT

Process Conditions for Melt Blown Fabrics in Second Phase

Although progress was made in producing good webs on the 20-inch MB line in the first phase of the STIR project, it had been observed that the TPU filaments were often traveling horizontally from MB die only a few inches or more, depending on air flow rates, before dropping vertically towards the floor. This observation coupled with the fact that relatively large MB fibers were being produced led us to believe that we had been going in the wrong direction with respect to spinneret hole diameter and air knife gap. Past experience at TANDEC has shown that with many high melt viscosity polymers such as polyesters and nylons, that the large hole die (0.018 inch hole diameter compared to the standard hole diameter of 0.0145 in.) and larger air knife gap of 0.090 inches actually results in finer fibers and softer webs. Therefore, in Phase 2 of the STIR project, the standard die tip with 0.0145 in. diameter holes and with an L/D of 8.5/1 and a hole density of 25 holes/inch was used. Also, the air knife gap on both sides of the nose tip was reduced to 0.030 in. and the die tip setback to 0.030 in.

Estane 58237, 58245 and 58280 pellets were obtained from Noveon and were dried for 12 hr at 100 °C in a Conair Franklin Compu-Dry CD 60 hopper dryer to a moisture content <0.02%. As shown in Table 2, a series of MB webs were produced with light, medium and heavy target basis weights of 80, 130 and 270 g/m^2, three different air flow rates and several different die-to-collector distances (DCD) ranging from 12 to 36 inches. Die temperature was held at 400°F, while air temperature in the die manifold was varied from 403 °F to 420 °F. Web collection speeds varied from 5 to 15 ft/min. Melt pressures after the metering pump varied from 670 to 1540 psig. Die air pressures

ranged from 3 to 10 psig, depending on the desired air flow rate to the die. The MB TPU webs were laminated with Kraft paper (to prevent deformation of webs from tension of winding) immediately after the collector, which was wound up with the webs during winding. Since the webs did not stick to the paper after cooling, a release finish is not needed on the paper, and much lighter paper may be used.

As shown in Table 2, the polymer throughput in trials 1.1 through 1.6 with Estane 58245 and in trials 2.1 and 2.2 with Estane 58237 was very low at 0.13 g/hole/min. in an effort to obtain the small fiber diameters. The throughput rates in trial 1.7 with Estane 58280 was 0.82 g/hole/minute and the polymer throughput rates in trials 2.3 with Estane 58237 and in trials 2.4 and 2.5 with Estane 58245 were much greater at 1.17 g/hole/min. A limitation of the study was the fact that the polymer throughput rates of the later trials were not determined by weighing web collected over a period of time (usually 5 minutes) because at these throughput rates (near maximum rpm of the 2-inch diameter screw and speed of gear pump), the dried batches of Estane polymers were being consumed too quickly to obtain 5-10 yards of samples for Heidi Schreuder-Gibson's Natick Laboratory, for Susan Hemphill at Noveon, and for retained samples to be tested at TANDEC. Still in retrospect, more care should have been taken to obtain measured throughput rates, even considering the rapid dynamics of the researchers in trying to maximize MB processing parameters. Nevertheless, the calculations of throughput based on the gear speed to actual throughput rate at 0.13 g/hole/min should be reasonably accurate since the Zenith Nichols gear pump (Single Steam Type H, Model HLB-5592, 10 cc/rev/port) is a positive displacement pump, although the melt densities of the different polymers would be expected to differ slightly. Another fact lending credibility to these polymer rates is that the gear pump/extruder system was operating at near maximum capacity. Under these conditions, the maximum throughput rate we have been able to achieve with high melt flow rate polypropylene (PP) is 0.9 g/hole/min, which should correspond with a throughput rate of 1.2 g/hole/min with the Estane polymer melts which have densities of 30-40% greater than PP.

Calendering of Estane 58245 Webs

Two representative MB webs produced from Estane 58245 (Samples 1.2 and 1.6) were calendered in the Kusters Calender on the Reicofil 2® Spunbond Line at TANDEC. The top steel roll has a raised diamond pattern with an area of 14.7% and the bottom steel roll is smooth. The actual surface temperature of both the upper and bottom rolls was 197 °F. Short samples of 3-5 yards were fed through the calendar by hand at a surface speed of 5 m/min. Two calendar nip pressures were used: 100 and 200 pounds/linear inch (PLI).

Determination of Fiber Diameter using Optical Microscopy

All microstructures from MB webs were examined by optical microscope using YS1-T Nikon at TANDEC, University of Tennessee, Knoxville. Image analysis process can be mainly divided to;

Image capture – Segmentation – Object detection – Measuring – Analysis

Fiber diameters using optical microscopy at 500 X magnification, were first measured in pixels. Ten fiber width measurements (care was taken not to measure the same fiber twice) were made from three web specimens (1 x 2 inch dimensions) taken diagonally from the two edges (2-3 inches from actual edge of web) and from the center of the web for a total of 30 diameter measurements per sample. A 50X UVFL Nikon lens and an image analysis program from the Scion Company, were used. Fiber diameters were converted from pixels to micrometers using the factor, 3.45 pixels/1 μm. Then imaging techniques such as thresholding, edge finding and region growing were employed. Finally software was used for automatically detecting optimal fiber images using the following macro-function steps:

1. Changing picture from color to black and white
2. The noise of picture is removed
3. Find the average brightness and contrast
4. Sharpen and characterizing image
5. By the user, the edge of fiber brightness and contrast are decided
6. From user's decision, fiber is searched by based on brightness and contrast, and
7. Fiber diameter is measured.

Results Obtained in Second Phase of STIR Project

The MB web properties obtained in the second phase of the STIR project are given in Table 3. MB web weights ranging from 79 g/m^2 (2.3 oz/yd^2) to 136 g/m^2 (4.0 oz/yd^2) were produced with Estane 58245, even at the low throughput rate of 0.13 g/hole/min in trials 1.1 through 1.6. The MB webs in this series were uniform in appearance and had a soft, dense structure. Considering the difficulty in producing small fiber diameters in melt blowing elastomeric polymers, surprisingly small average fiber diameters of less than 6 μm, as determined by the optical technique, were obtained with Samples 1.4, 1.5 and 1.6. Even more remarkable however was the fact that in the next series of runs **with Estane 58245, in trials 2.4 and 2.5, average optical fiber diameters of 7.89 and 5.24 μm, respectively were obtained at calculated polymer throughput rates of 1.17 g/hole/min.** As shown in Table 2, the air flow rates were increased as much as possible without causing excessive fiber breakage, but the mass ratio of polymer to air was 33.62 in trials 2.4 and 2.5, compared to 5.42 to 6.45 in trials 1.1 to 1.6. **It is indeed remarkable that such small fiber diameters were produced in the Estane 58245 trials 2.4 and 2.5 at such a high polymer throughput rate and correspondingly high polymer to air ratio. This finding, of course, greatly enhances the economic considerations in the MB processing of TPUs.** Furthermore, it was demonstrated that very high weight MB TPUs can be made with Estane 58245 in that web weights of 145 g/m^2 (4.3 oz/yd^2) and 273 g/m^2 (8.0 oz/yd^2) were obtain in trials 2.5 and 2.4, respectively. Although both webs were quite uniform in appearance and had good elasticity, these two webs had a firmer hand and were more dense, apparently due to less quenching at the very high polymer throughput rate employed.

In contrast to the MB webs produced with Estane 58245, the Estane 58280 and 58237 polymers resulted in coarser webs with a harder hand, although they were all highly elastic. Given the progress that was made in the melt blowing of Estane 58245, it is possible that better quality MB webs could be produced with the 58280 and 58237 TPU

polymers.

The measured fiber diameters using optical microscopy were characterized by the unit of pixels and uses image technique via computer. The effective diameter, based on air permeability, was calculated from the Equation (1), adapted from Tsai.[3)]

$$d_f^2 = \frac{D^2(1-c)^2 f(c)}{32} \tag{1}$$

Where, d_f= fiber diameter, c is web packing density, and D is pore size

$$f(c) = \frac{1.4 \times 4c}{-\ln c + 2c - 0.5c^2 - 1.5}$$

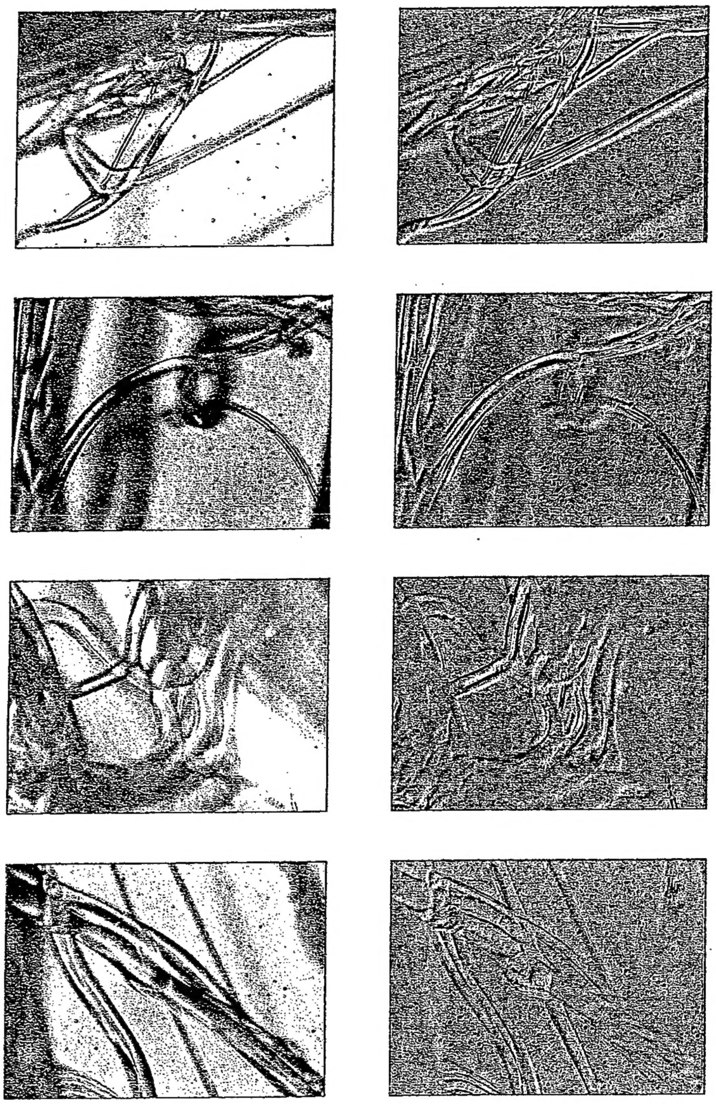
c = (volume of fiber)/(volume of media)
= (bulk density of the web)/(density of the polymer)

Although three different Estane TPUs were used in the second phase of the STIR project, and there is a limited number of data points, efforts have been made to show relationships between MB processing conditions and fiber diameters and web properties in Figure 2-1 through Figure 4. The correlation coefficient shown in each of the figures indicates the degree of linear association between a particular processing conditions and the resultant MB property. According to Anderson and Finn[4)], "the correlation coefficient has a possible range from -1 to +1." They note that the sign of r indicates the direction of association—positive for a direct association and negative for an inverse association, although "the absolute value of r is exactly 1 for a perfect linear relationship but lower if the points in the scatter plot diverge from a straight line." They further state that "statisticians would generally refer to a correlation close to zero as indicating *no correlation*, a correlation between 0 and 0.3 as *weak*; a correlation between 0.3 and 0.6 as *moderate*; a correlation between 0.6 and 1.0 as *strong*; and a correlation of 1.0 as *perfect*."

As shown in Figure 2-1, an almost perfect correlation (r = 0.98) exists between fiber size determined by the two different techniques is which is quite remarkable considering the fact that Effective Fiber Diameter (EFD) is based on air flow measurements and not from determining the physical size of fibers. Yet, EFD can be determined much more quickly than optical fiber diameters.

There is a moderate positive correlation between polymer melt pressure after the gear pump and optical fiber diameter, as can be seen in Figure 2-2(a), with the correlation coefficient, r, being 0.64, although the Estane 58245 has a wide scatter of fiber diameters in the low melt pressure region. A moderate correlation (r = 0.56) was also shown in

Figure 2-2(b) between EFD and melt pressure after the pump. However, there was much less scatter in the plots using EFD than with optically determined average fiber diameters in this figure and in Figure 2-3 through 2-6. In Table 2, it can be seen that Effective Fiber Diameters are generally much larger in magnitude than optically determined fiber diameters.



Sample 1.1: Resin 58245: 79 g/m^2

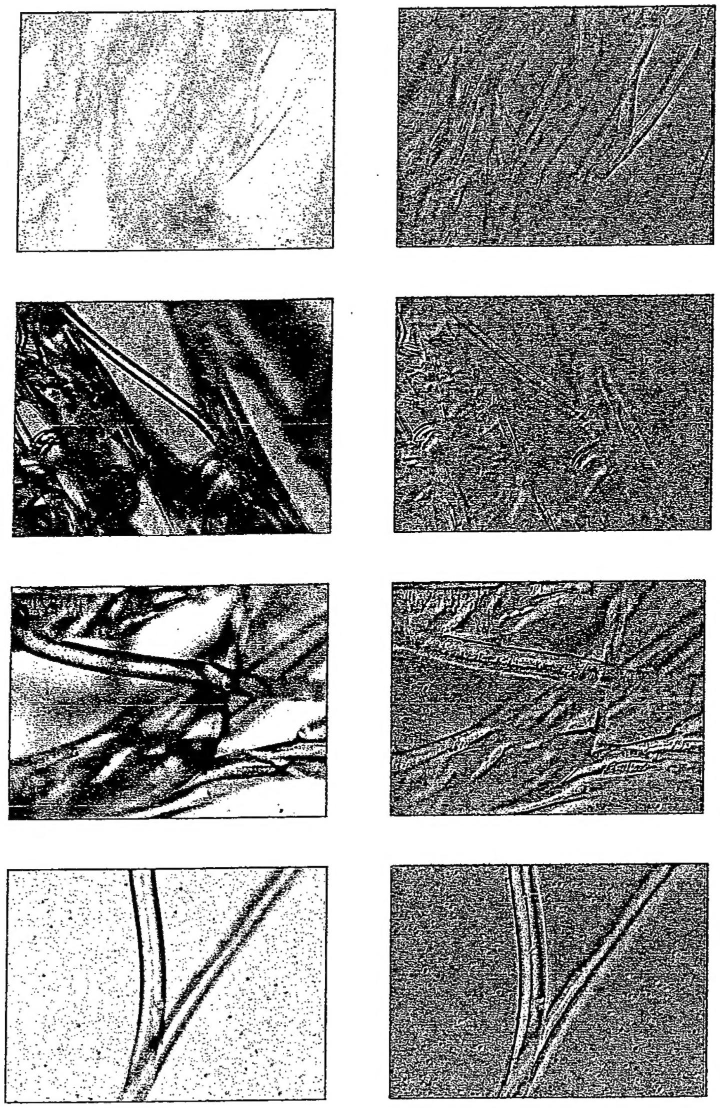
Sample 1.2: Resin 58245: 131g/m^2

Sample 1.3: Resin 58245: 124 g/m^2

Sample 1.4: Resin 58245: 136 g/m^2

(a) **(b)**

Figure 1-1. Optical microscope images and the corresponding processed images for sample 1.1, sample 1.2, sample 1.3 and sample 1.4
(a) Optical microscopy picture (b) Filtered and analyzed picture



Sample 1.5: Resin 58245: 97 g/m^2

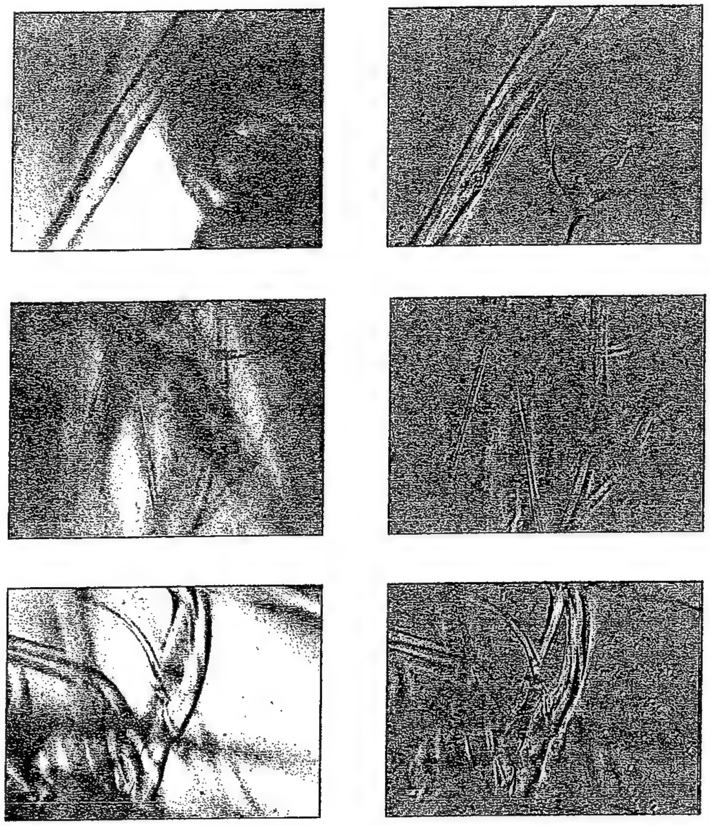
Sample 1.6: Resin 58245: 120 g/m^2

Sample 1.7: Resin 58280: 110 g/m^2

Sample 2.1: Resin 58237: 68 g/m^2

(a) **(b)**

Figure 1-2. Optical microscope images and the corresponding processed images for sample 1.5, sample 1.6, sample 1.7 and sample 2.1
(a) Optical microscopy picture (b) Filtered and analyzed picture



Sample 2.3: Resin 58237: 118 g/m^2

Sample 2.4: Resin 58245: 273 g/m^2

Sample 2.5: Resin 58245: 145 g/m^2

(a) **(b)**

Figure 1-3. Optical microscope images and the corresponding processed images for sample 2.3, sample 2.4 and sample 2.5
(a) Optical microscopy picture (b) Filtered and analyzed picture

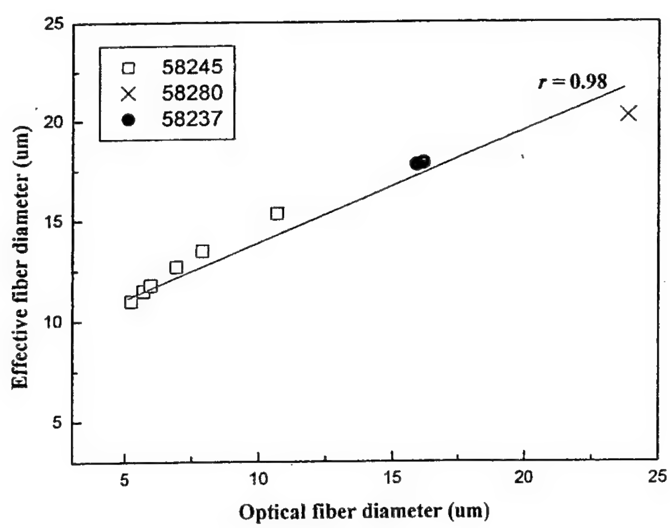
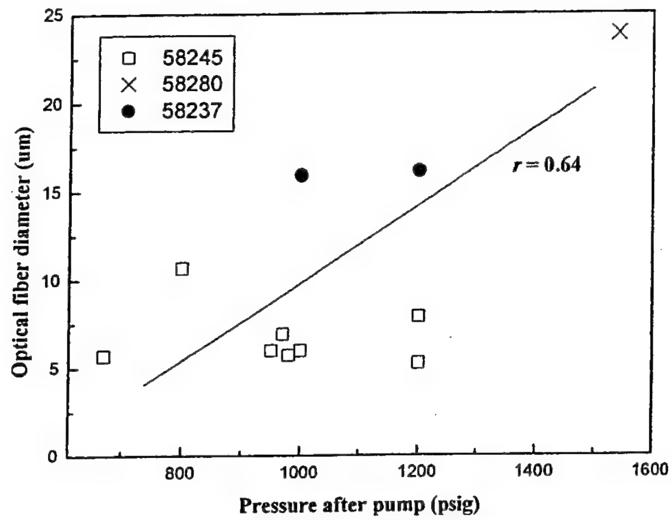
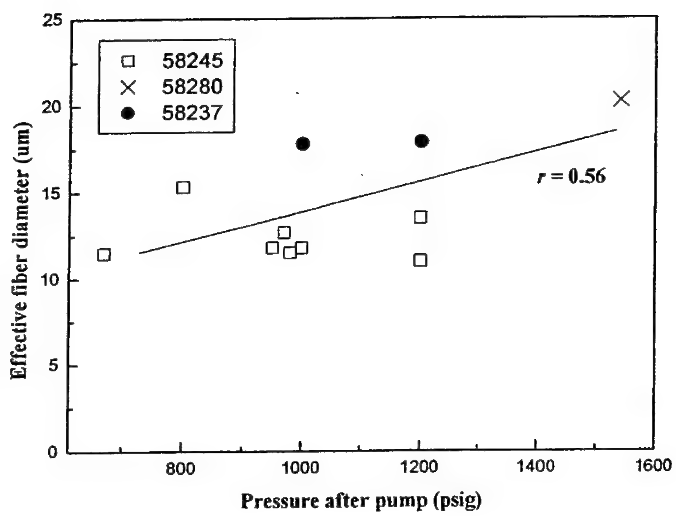


Figure 2-1. Relationship between optical and effective fiber diameter.



(a) Optical fiber diameter



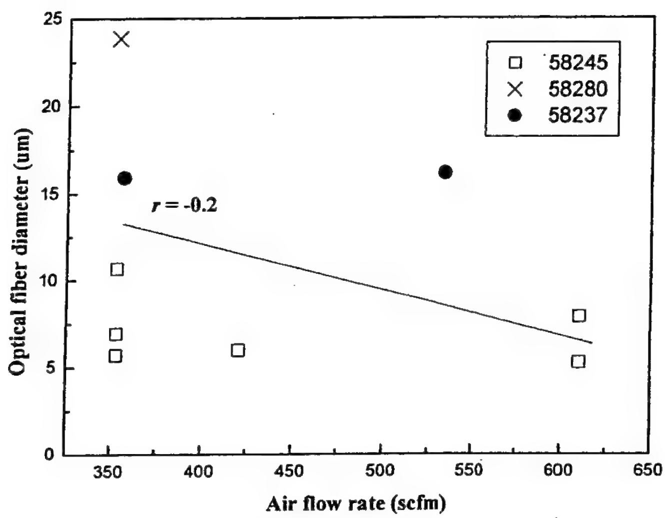
(b) Effective fiber diameter

Figure 2-2. Relationship between pressure after pump and fiber diameter

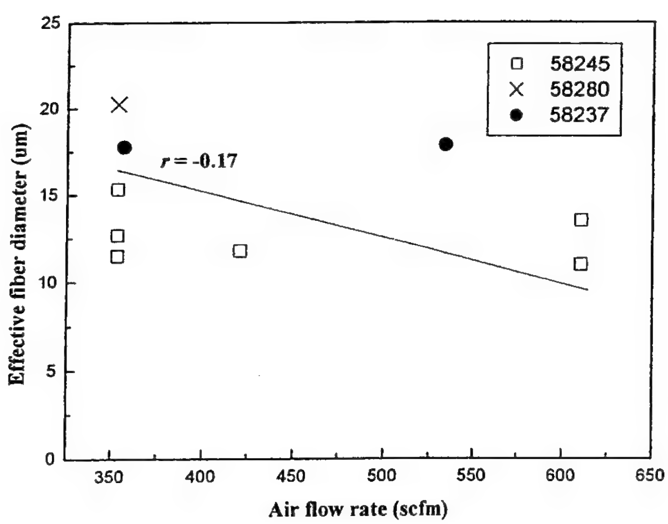
As shown in Figure 2-3, a weak negative correlation of about 0.2 was obtained between air flow rate (scfm/inch of die) to the die and fiber diameter, as determined by optical fiber diameter and EFD. In Figure 2-4, a weak positive correlation of about 0.3 was obtained between polymer throughput rate and fiber diameter (optical and EFD); whereas, there was a moderate positive correlation of about 0.5 between mass polymer to air ratio and fiber diameter as shown in Figure 2-5. In Figure 2-6, there is a weak negative correlation between actual air velocity at the die and fiber diameter, as determined by

both methods. In Figure 3-1, strong correlation ($r = 0.94$ with optical fiber diameter and $r = 0.89$ with EFD) was found between the TPU MB web properties fiber size and air permeability.

A weak positive correlation was found between the web thickness and air permeability in Figure 3-2 (a) and a weak negative correlation was shown between basis weigh and air permeability in Figure 3-2(b). A moderate positive correlation of 0.49 was obtained between basis weight and peak tensile load in Figure 4(a); whereas, essentially no correlation, 0.08, was shown between TPU basis weight and tearing strength in Fig. 4(b).

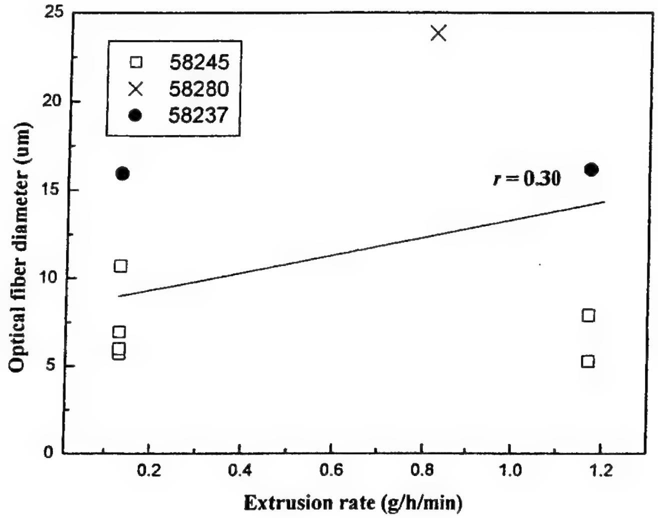


(a) Optical fiber diameter

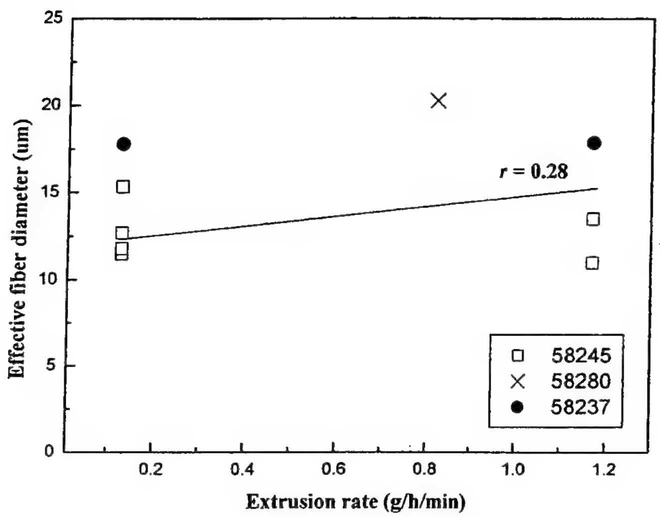


(b) Effective fiber diameter

Figure 2-3. Relationship between air flow rate and fiber diameter.

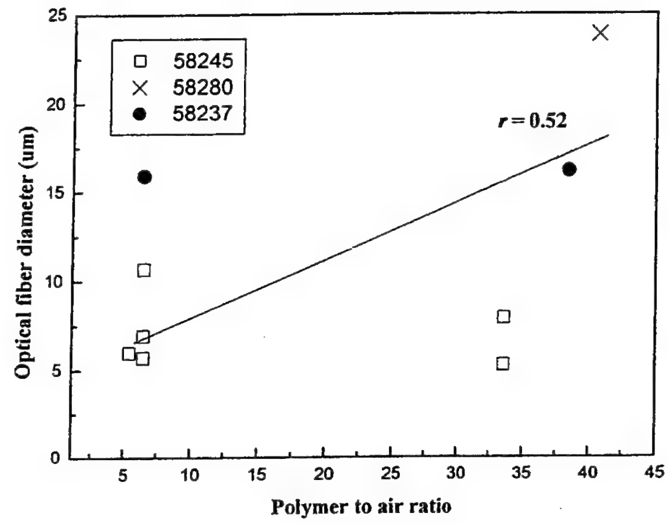


(a) Optical fiber diameter

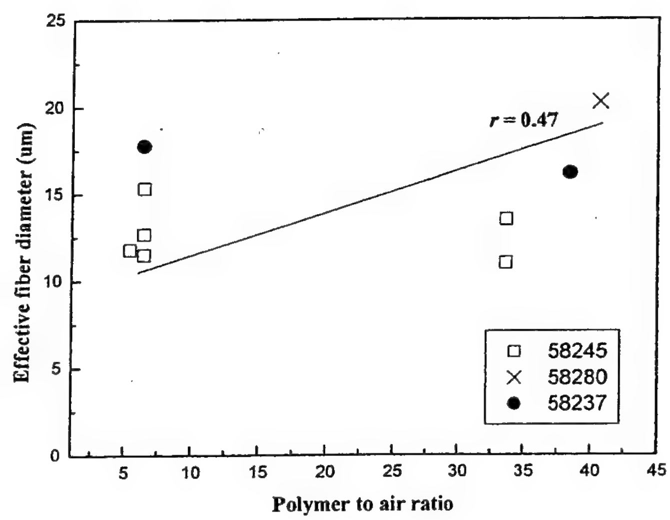


(b) Effective fiber diameter

Figure 2-4. Relationship between extrusion rate and fiber diameter.

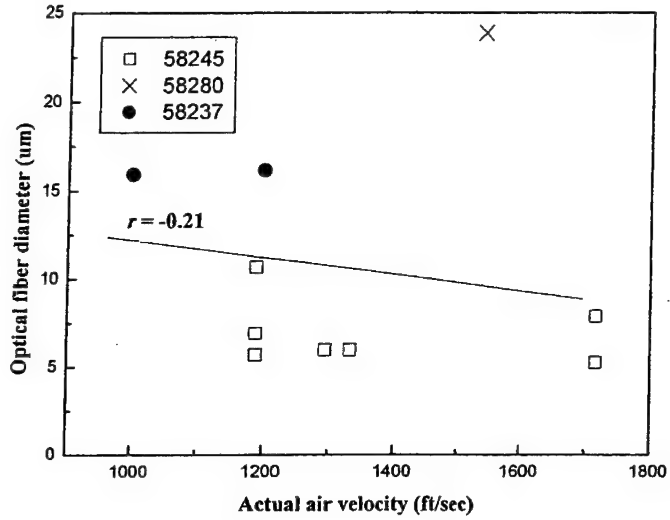


(a) Optical fiber diameter

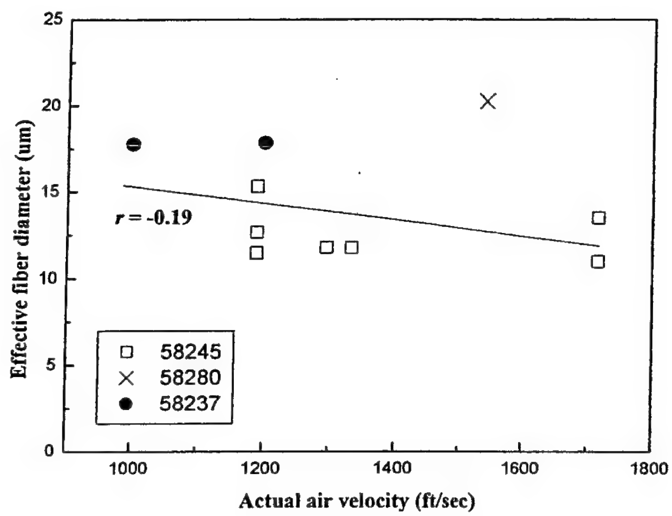


(b) Effective fiber diameter

Figure 2-5. Relationship between polymer to air ratio and fiber diameter.

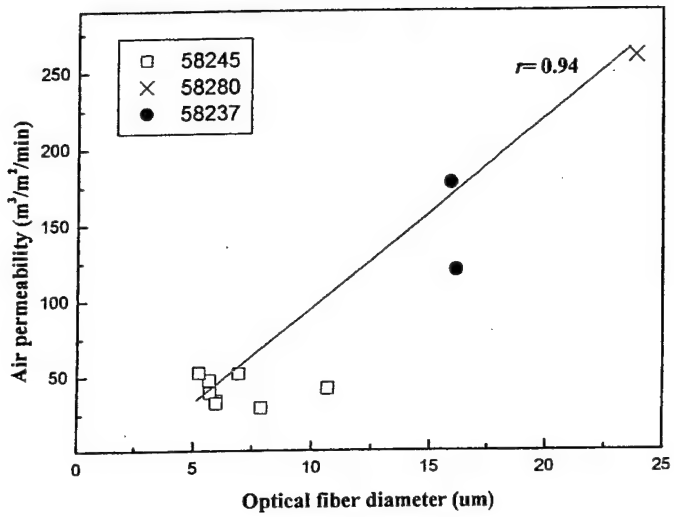


(a) Optical fiber diameter

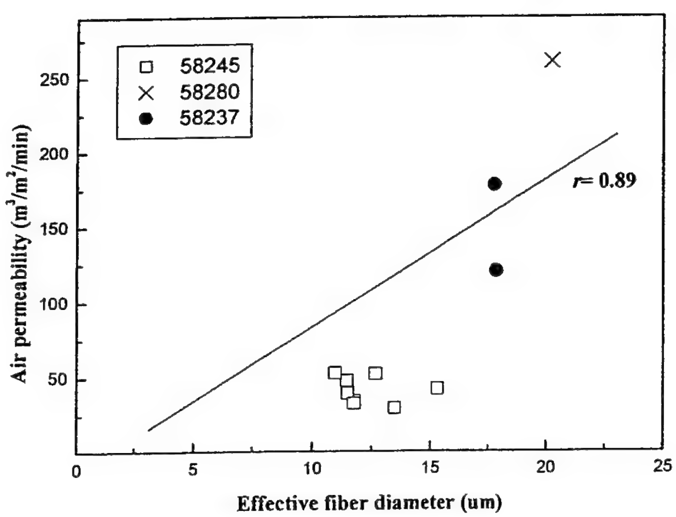


(b) Effective fiber diameter

Figure 2-6. Relationship between actual air velocity and fiber diameter.

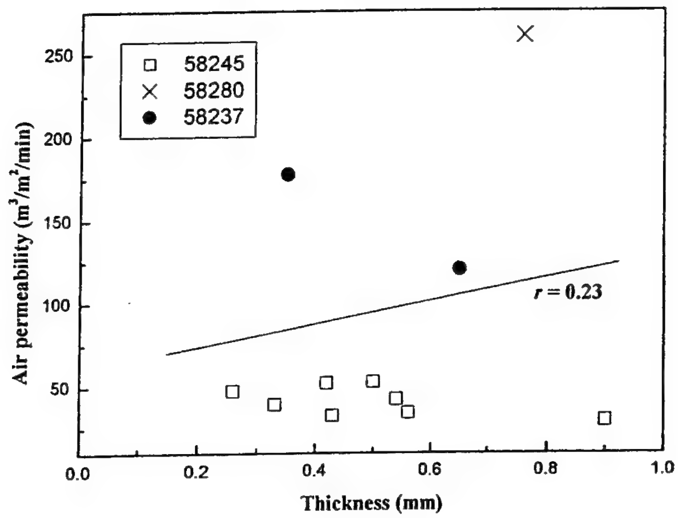


(a) Optical fiber diameter

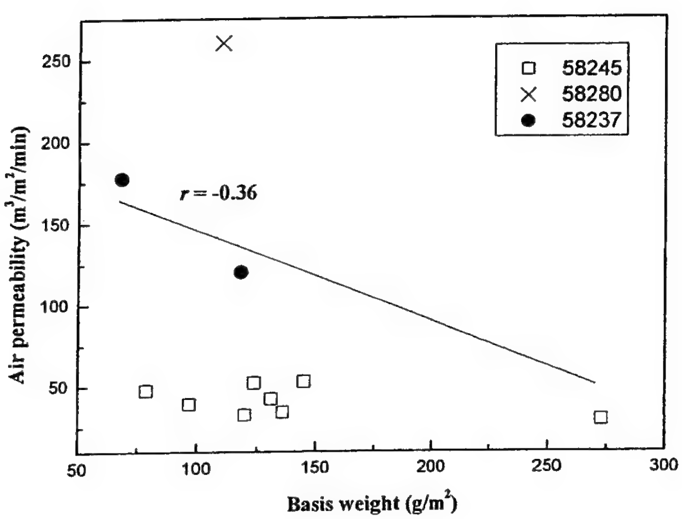


(b) Effective fiber diameter

Figure 3-1. Relationship between air permeability and fiber diameter.

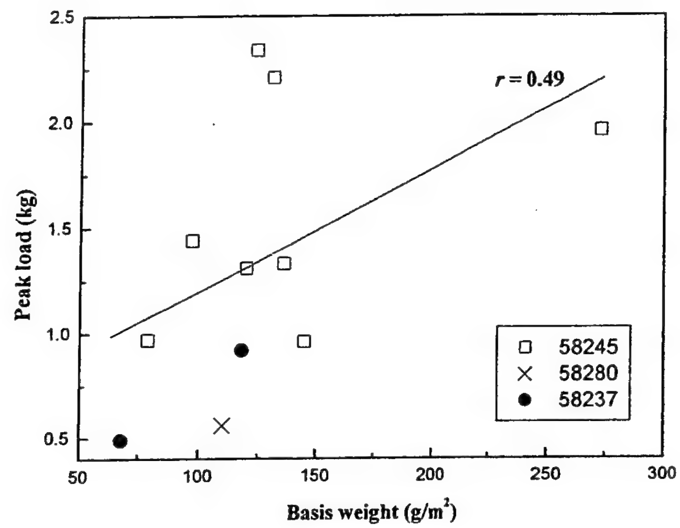


(a) Thickness

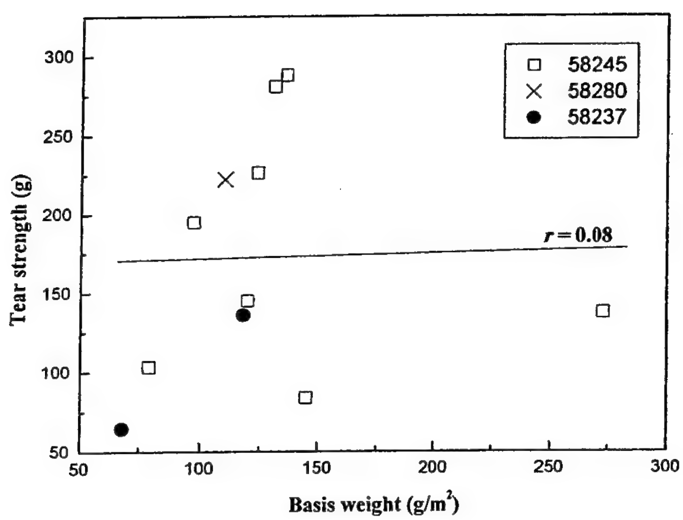


(b) Basis weight

Figure 3-2. Relationship between air permeability and web basis weight.



(a) Tensile strength



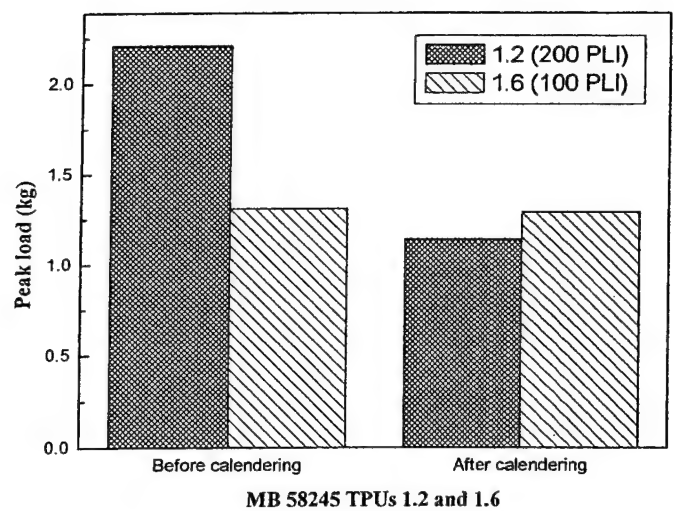
(b) Tear strength

Figure 4. Relationship between web tensile and tearing strength and basis weight.

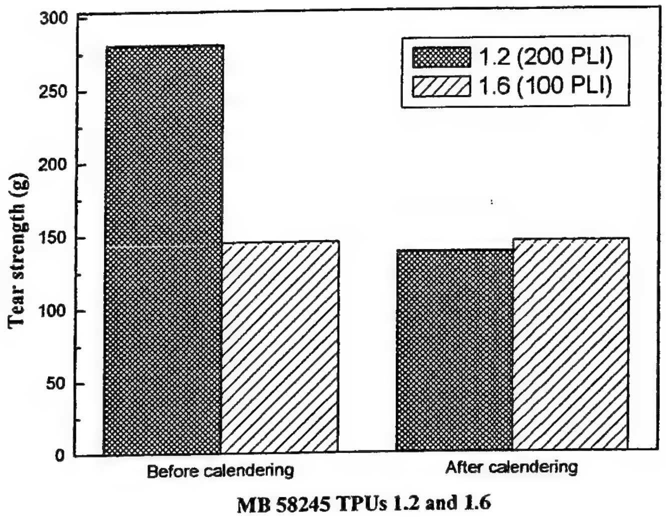
In order to determine the effect of thermal point calendaring on the strength of MB TPU webs, two Estane 58245 MB samples, 1.2 and 1.6, were subjected to thermal point calendaring. As shown in Figure 5 both tensile and tear strength remained about the same as the non-calendared samples at a calendaring pressure of 100 PLI but decreased notably at 200 PLI. This shows that MB TPU webs may be given a pattern or texture by calendaring without diminishing the strength properties. Other applications in which calendaring of MB TPU webs may be desirable could be to better lock in particulate additives such as activated carbon fibers, powders or fibers or to laminate the MB TPU webs to other structures.

CONCLUSIONS—First and Second Phases

1. Melt blown (MB) fabrics from Estane 58245 thermoplastic elastomer exhibited the best balance of weight, strength and fiber diameter as well as good MB processing performance.
2. Essentially a perfect correlation was shown between average fiber diameter as determined by optical microscopy and between Effective Fiber Diameter (EFD) as determined from air flow measurements, although the EFD values were generally much greater than fiber size obtained from microscopy.
3. Strong positive correlations were obtained between fiber diameter and melt pressure after the pump and moderate positive correlation was sown between fiber diameter and mass polymer to air ratio.
4. Strong positive correlation was found between fiber size and air permeability; whereas, a moderate positive correlation was found between MB TPU basis weight and peak tensile load, and essentially no correlation was found between basis weight and tearing strength.
5. The calendering process does not reduce the tensile and tearing strength at low bonding pressure but reduces the strength of the MB TPU web with higher bonding pressure, which shows MB TPU webs may be calendered as needed for better containment of particulate additives or for lamination.



(a) Tensile strength



(b) Tear strength

Figure 5. Influence of the calendaring on tensile and tearing strength of MB TPU webs.

THIRD PHASE OF STIR PROJECT

Also, with continuous input from the above trials, the PI (Wadsworth) supervised a MB equipment design consultant, Bernard L. Beals, Beals Consulting, 108 Vantage Lane, Hamilton, MT, in the design of a high performance 10-inch MB line (more suitable for polycondensation polymers with additives, such as TPU's) with interchangeable polymer melt die coat hanger sections for polymers and additives with a wide range of melt viscosities. The design of MB line using a twin-screw extruder and different geometries of web collectors was also studied with the intent of maximizing the MB processing and web performance properties of TPUs as chemical protective liners. Through the efforts of Wadsworth a twin screw extruder was located at Shell Chemical Company, Houston, TX which was available for donation to the University of Tennessee (UT) for this program. Beals visited Shell Chemical in August 2001 and confirmed that the extruder was indeed ideally suited for the melt blowing process. The Berstorff Model ZE40 40 MM Twin Screw Extruder valued at $250,000 was donated and shipped to UT in February 2002.

The twin-screw extruder will allow chemical additives and enzymes which are heat sensitive to be introduced near the delivery end of the extruder, thereby greatly improving their odds of not being deactivated during MB production. Also, it may be feasible to compound in the metal oxides during MB production, thereby, eliminating the need for a separate compounding step. Furthermore, since the twin-screw extruder is vented for water vapor escape, which may make the pre-drying step less critical since residual moisture will be continually vented from the extruder.

Under Wadsworth's supervision, Beals designed a 10-inch MB die suited for melt blowing TPUs, but unfortunately the SBIR Proposal Number A012-0921 for Topic A01-030 "Selectively Permeable Elastomeric Membranes for Protective Clothing" submitted by USPA Inc., Knoxville, TN, in cooperation with UT was not funded. If funded, this project would have provided funds for the rental of off-campus laboratory space and for auxiliary equipment to install the 10-inch MB line using the twin-screw extruder donated by Shell Chemical. Unfortunately, the SBIR was not funded and the construction and installation of the high performance melt blown line must be postponed until sufficient funding can be obtained. In the meantime, a proposal is being prepared based on the accomplishments of this STIR project, which will be submitted to the U.S. Army Soldier Biological Chemical Command, and to other DOE agencies, for possible funding beginning October 1, 2002, to install the twin-screw extruder on the 20-inch Accurate Products melt blown line at TANDEC so that further improvements can be made in the melt blown production of TPUs and in the promising development of reactive MB TPUs for Chemical Protective Liners.

OUTCOMES OF STIR PROJECT

In addition to making notable advances in the melt blowing of TPUs, designing the next generation of high performance melt blown equipment for producing elastomeric melt blowns and other high performance materials, and obtaining the donation of the twin-screw extruder from Shell Chemical valued at $250,000, the following conference papers have resulted from this work:

1. The research findings from the preliminary study performed before the STIR project was initiated were presented by Larry C. Wadsworth at the joint INDA/TAPPI International Nonwovens Technical Conference (INTC 2001) in Baltimore, MD during September 5-7, 2001[1)]. The authors of this paper were Larry C. Wadsworth, Christine (Qin) Sun, Dong Zhang and Rongguo Zhao of TANDEC, University of Tennessee and Heidi L. Schreuder-Gibson and Phil Gibson, U. S. Army Natick Soldier Systems Center.
2. A second paper is being presented by Heidi L. Schreuder-Gibson titled "Effect of Filter Deformation on Filtration and Air Flow for Elastomeric Nonwoven Media" at the 15th Annual Conference and Exposition, American Filtration and Separations Society, Galveston, TX, April 9-12, 2002[5)]. For this paper MB TPU Sample 1.5 (Estane 58245) with a weight of 97 g/m^2, which was produced in the Second Phase of this STIR project, was used as substrates onto which TPU nanofibers were deposited by an electrospinning process at the Natick Soldier Center. The authors of the paper are Heidi Schreuder-Gibson and Phillip W. Gibson, Natick Soldier Center; Larry C. Wadsworth, University of Tennessee; and Susan Hemphill and Joe Vontorcik, Noveon, Inc., Brecksville, OH.
3. A third paper has been accepted for presentation by Larry C. Wadsworth titled "Improvements in Melt Blown Thermoplastic Polyurethane Fabrics for Elastic Military Protective Chemical Liners," at the International Nonwovens Technical Conference (INTC 02) cosponsored by INDA and TAPPI at Atlanta, GA during September 24-26, 2002[6)].

Furthermore, when Dr. Heidi Schreuder-Gibson, STIR project manager, noted to Dr. Wadsworth that she might require additional protection offered by activated carbon for the MB TPU chemical protective liners, Wadsworth recommended that she contact Moldex Metric Inc., Culver City, CA, a firm which commercially produces MB PP respirators, which are heavily loaded with activated carbon particles during MB production by blowing the carbon particles into the MB air/fiber stream between the die and collector. This report will be used by Moldex Metric prepare carbon-loaded MB TPU fabrics under contract for Dr. Heidi Schreuder-Gibson.

ACKNOWLEDGEMENTS

The authors wish to express their gratitude to Susan Hemphill of Noveon (formerly B. F. Goodrich) for technical advice and a complimentary supply of Estane 58237, 58245, 58238 and 58280 for this study. The funding received from the Army Research Office through Agreement No. DAAD19-01-1-0732 is most appreciated.

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Table 1. Melt Blown Processing Conditions and Web Properties in Phase 1 of STIR Project

Description		Weight (g/m^2)	DCD (inches)	Speed (fpm)	Die-Temp. (Deg. F)	Air-Temp (Deg. F)	Pump (psig)	Die Air Pressure (psig)	Thickness (mm)	Air permeability (m^3/m^2/min-125Pa)	Effective (Optical) fiber size (um)
Resin	Sample No.										
58237	1.1(6")	-	19	5	380	388	522	6.5	NA		
58237	1.2(6")	-	19	15	380	384	321	10.0	NA		
58237	1.3(6")	150	19	15	380	384	276	5.0	0.616	61.9	12.86 (6.91)
58245	1.4(6")	-	19	15	380	402	505	4.0	NA		
58238	1.5(6")	-	10	5	380	388	541	1.0	NA		
58280	1.6(6")	-	17	4	380	413	-	2.0	NA		
58280	2.1(20")	71.25	10	22	390	416	3700	3.0	0.465	197.6	19.14 (19.13)
58245	2.2(20")	17.5	10	8	390	418	3700	3.0	0.120	332.1	11.29 (5.42)
58280	2.3(20")	100	10	13	390	418	3700	3.0	0.714	349.8	19.57 (20.31)

Table 2. Melt blown processing conditions for Estane TPUs in Phase 2 of STIR Project

Description		Weight (g/m^2)	DCD (inch)	Speed (fpm)	Polymer Thru-put		Press after pump (psig)	Die-Temp. (°F)	Air-Temp. (°F)	Actual air velocity (ft/sec)	Air flow rate (scfm)	Polymer to Air Ratio
Resin	Sample No.				Gear Pump (rpm)	Extrusion Rate (g/h/min)						
58245	1.1	79	36	15	7.5	0.13	670	400	409	1189	353.51	6.45
	1.2	131	36	5	7.5	0.13	800	400	405	1189	353.51	6.45
	1.3	124	36	5	7.5	0.13	970	400	403	1189	353.51	6.45
	1.4	136	36	5	7.5	0.13	950	400	410	1295	420.94	5.42
	1.5	97	12	5	7.5	0.13	980	400	405	1189	353.51	6.45
	1.6	120	19	5	7.5	0.13	1000	400	405	1333	420.94	5.42
58280	1.7	110	13	NA	47.5	0.82*	1540	400	403	1153	353.51	40.68
58237	2.1	68	12	5	7.5	0.13*	1000	400	407	1143	356.80	6.39
	2.2	NA	18	5	7.5	0.13*	1130	400	411	1320	432.96	5.27
	2.3	118	14	5	67.5	1.17*	1200	400	418	1597	533.96	38.43
58245	2.4	273	22	5	67.5	1.17*	1200	400	420	1716	610.39	33.62
	2.5	145	22	8	67.5	1.17*	1200	400	420	1716	610.39	33.62

Nozzle hole diameter: 0.0145in, Hole L/D: 8.5/1 Air gap: 0.030in

Number of hole/20in: 601 (25 holes/in.) Set back: 0.030in

Zenith Nichols, Type H, Model HLB-5592, 10 cc/rev/port

*Calculated thru-put rates based on gear pump rpm * Webs were laminated to roll paper between collector and winder

Table 3. Melt blown web properties for Estane TPUs in Phase 2 of STIR Project

Description		Weight (g/m^2)	Thickness (mm)	Tear Strength (g)	Air Permeability ($m^3/m^2/min$)	Effective(Optical) fiber size (um)	Touch and structure of surface	Tensile strength	
Resin	Sample No.							Peak Load (kg)	Peak Elong. (kg)
58245	1.1	79	0.261	103.3	47.58	11.47(5.68)	S, D	0.97	331.0
	1.2	131	0.538	280.7	42.18	15.32(10.67)	S, D	2.21	443.5
	1.3	124	0.422	226.3	52.06	12.68(6.92)	S, D	2.34	442.8
	1.4	136	0.561	287.8	34.07	11.79(5.98)	S, D	1.33	233.4
	1.5	97	0.333	194.7	39.31	11.50(5.71)	S, D	1.44	418.8
	1.6	120	0.430	145.0	32.54	11.77(5.97)	S, D	1.31	266.6
58280	1.7	110	0.759	222.3	260.23	20.25(23.85)	H, R	0.56	218.4
58237	2.1	68	0.353	64.5	177.54	17.77(15.91)	H, R	0.49	43.2
	2.2	NA							
	2.3	118	0.650	135.9	120.23	17.86(16.15)	H, R	0.92	78.5
58245	2.4	273	0.900	137.2	29.28	13.48(7.89)	H, D	1.96	208.4
	2.5	145	0.498	83.7	52.70	10.98(5.24)	H, D	0.96	154.8

H: Harsh surface S: Soft surface
R: Rough structure D: Dense structure